Notebook Series -- Notebooks by Experimenters Other Than Edison
Phonograph Record Experiments
Miscellaneous Disc Composition Books
Notebook, N-11-04-17.2

This notebook was used by W. Jones in 1911 for notes on experiments involving the shellac used to coat the Edison disc records that would be put on the market in 1912. The object of the experiments was to reduce blisters, smears, and other imperfections. The notes indicate that A. Petit (either Ademor N. or Albert O.) collaborated with Jones on the new disc records. One note by Edison has been inserted into the book. The front cover is labeled "Shellac # 2064" and "W. Jones, Chemical Laboratory." The pages are unnumbered. Approximately 50 pages have been used.

N lac 3 9 6/10 x 3 = 18

V V. 1 3

½ SV 1/10 3/10

200 augms V, Bl.

½

1/10 x 16

1.6

Acme Co.,

MFG. STATIONERS,
69 FULTON STREET,
NEW YORK.

45009

N-11-04-17.2

1911

April 17

20 gm shellac T.L.+Co B+I superior
35 cc Borax
Boiled ten minutes.
Cooled w. water
Diluted to 2 times volume with cold water and filtered off the solidified wax which rose to the surface. Difficult to filter.
Precipitated the shellac acid by dilute HCl. (Hydrated acid) Light pink flocculent ppt. Dried to brown
Filtered off by suction.
Made following experiments on resultant after drying on steam bath.
Fused some and while plastic pulled it like molasses candy. This gave a good tough article with tetrachlor naphthalene but condensed too readily. Shellac from which the wax has not been removed does not condense so readily. (Condensation may be prevented by the wax??)

over

Made up a one pound batch.
180 gms Comil Borax
1½ lbs shellac
fft as before and pulled the resulting shellac.
Made following trials with 1 part tetrachlornaphthalene + 3 parts shellac + 1lb following:
Anidine: kept liquid till ambine has evaporated.
Pyridine: little better
Diphenylamine: N.G. } brittle
Naphthylamine: N.G.
Xylidin: to rapt
Dimethylaniline: same as pyridin
Toluidine: best yet
Oil of Amber: This keeps the mass liquid longer than any of the others. Combined with toluidine it gives the best results. The melt should be kept on the heat till the gases are almost all given off when the mass will be fairly liquid. Increase in the oil amber makes the resultant mass very elastic.

Oil of Amber + pyradine: This is even better than the toluidine which is slightly brittle.
The oil of amber + tetrachlornaphthalene + pyridine reacted to form a tarry and small pieces of shellac added. Then till frothing ceases and let cook till a drop on paper does not show a grease ring.
Residue from distill. of Amber Oil: This did not give good results.
Crude Amber Oil: Fair but not not hard enough. After heating some minutes coagulate as usual.
Rosin Oil (1st Run): Good but does not keep liquid long enough
Venice Turps: N.G.
Anthraquinone: N.G.
Quinoline: Too soft. Bubbles
Meta-Toluidin: No better than the other bases. Addition of acetanilide has no effect

Tried fixing the shellac acids with metallic salts as fillers.
Lead Acetate = too brittle
Tried following:

I

1 gm diphenylamine
3 gm tetrachlornaphthalene
9 gm ord. shellac
Little too soft. Keeps liquid
very few bubbles.

II

1 gm diphenylamine
3.3 gm tetrachlor
10 gm ord. shellac
Better. Little too soft.

III

1 gm diphenylamine
3 1/3 gm tetrachlor
11 gm shellac acids and alcs. ordinary
Better with ordinary N.G. with acids.

IV

1 gm m-tolylenediamine
3 1/3 gm tetrachlor
11 gm shellac (ordinary)
Absolutely N.G.
Won't fuse together

V

1 gm paraphenylenediamine
3 1/3 gm tetrachlor
11 gm shellac
N.G. same as IV

VI

1 gm hexamethylenetetramine
3 1/3 gm tetrachlor
11 gm shellac
Very hard and fuses fairly well but does not stay liquid long enough.

VII

1 gm tolidine (pure base)
3 1/3 gm tetrachlor
10 gm shellac
N.G. Condenses at once

VIII

Same as VI with 3.3 tetrachlor
+ 10 shellac
N.G. condenses.

IX

1 gm diphenylamine
4 gm tetrachlor
12 gm shellac ord. Good
added 2 gm more shellac

The resultant masses from formulae I, II, III, and IX on previous page became very hard on standing over night and were a little too brittle. No. I was the best. They stood heat well. They all seem to decompose much less on the subsequent remeltings. Tried following to secure more softness:

I A
1.2 gm diphenylamine
3 gm tetrachlor naphthalene
9 gm ord. shellac

I B
1.3 gm diphenylamine
3 gm tetrachlor
9 gm ord. shellac

I C
1.1 gm diphenylamine
3 gm tetrachlor
9 gm shellac

I D
1.05 gm diphenylamine
3 gm tetrachlor
9 gm shellac

I E ✓
1.5 gm diphenylamine
3 gm tetrachlor
9 gm shellac

These five preparations were remelted 7 times and allowed to stand over night. Became almost too hard. E is best

Tried α-naphtol in place of the diphenylamine in formula I B. Kept liquid, gave very few bubbles and stood remelting 7 times.

Trichlorquinone: N. G. Condensed.

Tried pure shellac acids in place of shellac in all the preceding formulae and the mass crumbly, condensed sooner or later, and decomposed violently.

Continued work with diphenylamine:

I F
1.7 gms diphenylamine
3 gm tetrachlor
9 gm shellac

over

I G

2 gms diphenylamine
3 gm tetrachlor
9 gm shellac

I H

2.5 gms diphenylamine
3 gm tetrachlor
9 gms shellac

I J.

3 gms diphenylamine
3 gms tetrachlor
9 gms shellac

I K

3.5 gms diphenylamine
3 gms tetrachlor
9 gms shellac

Made up a 1/2 lb batch for trial using formula
1.5 oz diphenylamine
3 oz tetra
9 oz shellac
Strained thru cheese cloth at 130° C. Kept at 120° C for a few minutes and poured. Remelted, kept at 110-115° C + poured. Made 11 oz

I M

Tried the following modifications:
1.1 gm diphenylamine
0.1 gm Borneol
3 gms tetra
9 gms shellac

I N

1 gm diphenylamine
1 gm monobromated camphor
3 gms. tetra
9 gms shellac

I O

1 gm diphenylamine
1.5 gm Borneol
3 gm tetra
9 gm shellac

Camphors too volatile.

OVER

Started investigation of the free lac acids.

The resin acid exists in the shellac as a monobasic acid. This polymerizes on boiling with caustic alkalies to a tribasic acid. (Benedict + Ulzer Monats. 5 528)

Obtained a quantity of the tribasic acid as follows:

50 gm lac acid
15 gm NaOH
1 l. H_2O

Boiled for 2 hrs. On precipitating with dil H_2SO_4 a viscous mass separated. Extracted this w. ether. Distilled off ether. Residue = the impure tribasic acid.

Benzoylated some pure shellac acid by saponifying with NaOH and then boiling this solution under a reflux with benzoyl chloride. At the last a little KI is added to ppt all the acid. After washing well a rubbery mass is obtained becoming very adhesive on heating and with a slight odor of benzoic acid.

The benzoylated acid condenses after fifteen minutes heating.

Tribasic acid, previous page:

Boiled up the residue with NaOH + added CrK_2O_4 and $MgSO_4$. A flocculent reddish ppt. came down. This ppt. insol. in alc. On filtering a dark red filtrate was obtained containing a resinous substance very sol. in alc. Its Mg. salt was even very sol. in alcohol. On ppting with acid and extracting with ether a resinous light yellow substance was obtained from the filtrate.

The Mg salt remaining on the filter was made into a suspension w. H_2O in a sep. funnel, acidified and extracted w. ether. A dark resinous substance was obtained. A small amt of a black adhesive substance would not dissolve in the ether.

The filtrate on acidifying and shaking out with ether yielded

a fairly liquid dark red substance. Both these resinous substances condense almost immediately on heating.

Prepared benzylated and acetylated lac acid by acting upon the Na salt in the cold with benzyl and acetyl chlorides respectively. They both are very adhesive pasty brown substances which on melting decompose rapidly.

Attempted to decolorize some of the lac acids by boiling all day with blood carbon in abs. sol. and then fit the filtrate with H_2O + boiling off the alc. The acids still of a light brown color although considerably lighter than before.

Call the tribasic acid which stayed on filter as insol. Na - salt, α-resin and the acid which passed into filtrate the β-resin.

α-resin slightly sol in alc.
β-resin very easily to clear dark red sol.

Using the decolorized acid made following:

4.5 gm acid
1.5 gm tetra
0.4 gm diphenylamine

Condensed almost immediately

Tried following with lac acid:

Dinitrobenzol	N. G.
Resorcin	N. G.
Phenoresorcin	N. G.
Perchlorbenzol	N. G.
Dioxynaphthalene	N. G.
Naphthalene	Better
Acetotoluidin	N. G.
Azobenzol	N. G.
Hydrazobenzol	N. G.
Anthracene	N. G.
Phenanthrene	N. G.
α-mononitronaphthalene	N. G.
Acetanilide	N. G.
p-nitroacetanilide	N. G.
Benzidin	N. G.
Amidoazobenzol	N. G.
Tetramethyldiamidodiphenylmethane	N. G.
Hexachlorethane	N. G.

Warmed a portion of snowbairic acids with CHCl3 out from three portions till all the alcohol soluble acid had dissolved. Filtered and obtained a dark red filtrate and a gelatinous brown mass on the filter. The latter on drying became a hard dark brown powder, which carbonized at 150°C without melting nor would it dissolve in tetra or in diphenylamin on heating. This residue was insol. in

alcohol HCl slight
amyl alcohol H_2SO_4 slight carb.
chloroform
ether Was sol. in strong
benzol alkalies.
acetone
benzin

Boiled some alcoholic shellac sol. with bone black, filtered, ppted the filtrate w. ~~ale~~ water and 2 drops HCl. Filtered, evaporated filtrate to dryness, extracted residue with ether & on evaporating obtained a very small quantity of a crystalline substance colorless and crystallizing in fern pattern. Probably laccic acid. Too small amt. to do anything with.

Passed anhydrous NH_3 through a borax sol. of shellac. Changed to a perfect jelly. This jelly solubly in hot water and hot alc. with great ease. Acids ppt. a flocculent light brown resinous substance. This when dried and melted condenses and behaves like the normal lac acids.
The ammonia jelly when melted first discolors in its own water and then decomposes.

The Pb, Mn, Mg, Na, Li, Al Zn, Fe, salts all decompose with frothing.

On leading anhydrous NH_3 through an alcoholic sol. of tribasic β-resin and distilling off the alcohol, a resin is obtained which on heating for some time flows perfectly limpid with absolutely no decomposition and apparently stays liquid for about 2 hrs with steady heat. Resin too soft.

The solution becomes quite warm on passing the NH_3.

The monobasic acid treated in similar manner does not become warm but turns to a deep cherry red color, dont on evaporating and heating condenses almost at once.

If the alk solution is boiling while the gas is conducted thro it the resin is much softer and very soluble in H_2O. The monobasic lac acid acts similarly.

On conducting SO_2 thro the tribasic acid a resin is obtained in all respects similar to the above.

Prepared another batch of lac acids but washed them better than the last and did not pull them while soft.

These acids behave much better than the last as regards condensation, although they condense quicker than the raw shellac.

Tried Aniline Violet Base, Induline Blue + Black with lac acids and with shellac. Made very tough masses with acids but they decomposed too much. With shellac they made the mass too brittle

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Using lead stearate and diphenylamine a shellac mass can be kept liquid at 140°C for 35 minutes. (Not long enough)

Separated raw shellac as follows:

Raw shellac

1. Soluble in cold alcohol + ether
2. " " " " not in ether
3. Insoluble in the usual organic solvents.
4. Crystalline wax (from hot C$_6$H$_6$)

The resins 1, 2, 3, were very easily decomposed.

No. 1 fused and almost immediately condensed

No. 2. Decomposed with intumescence

No. 3. Carbonized without fusion

The wax melted readily to a limpid fluid.

The resin No. 1 when fused from coloring matter was no better

Separated the lac acids.

1. sol. in cold alc + ether
2. " " " " insol in ether
3. Insol.

These resins behaved identically with those from raw shellac.

Raw shellac

On further trial with a sand bath and constant temperature, the resin No. 1 kept liquid for 1 hr + and solidified with tetra to a very hard mass having a cut like horn. Good + tough except the bubbles which are very difficult to get rid of.

Tried the following as solvents in hopes to get rid of the bubble.

Fused oil — Best. Few bubbles

Dil. acetic acid — Condenses

Dil. HCl. — Does not dissolve.

Even on precipitating the alcoholic sol. of resin # 1 with water and drying 2 hrs, subsequent melting does not get rid of the bubbles.

On warming shellac with fused oil a large part goes into solution. Filter, distil off main portion of fused oil and heat on sand bath with tetra and lead stearate. Very few bubbles form and keeps only limped

On chlorinating alc. sols. of mono- and tribasic acids and resid #1, evaporating etc, the following results obtained:

Tribasic acid: Heated on sand bath three days. Became more viscous each day till it finally condensed. Never got harder than tar.

Monobasic acids: condensed in a few hrs. Never got harder than molasses

Resin #1: Became soft + viscous after few hrs. heating.

In all cases the solutions were bleached to a great extent

On trying the fusel oil method on a larger scale it appeared impossible to get rid of the bubbles, traces of fusel oil being very difficult to remove without old heating

Different Grades of Gum Lac

Made up a batch with 3 to 1 tetra-lac of following:

Rogers + Pyatt

~~T.A. Pure 15½ #: Kept at 125° five minutes and foamed. Bubbles showed signs of diminishing as heating proceeded. Not quite so fluid as regular. Not Bad.~~

(A)

R+P. Pure Garnet: 15½ #. Had to keep at 135° in order to keep sufficiently fluid. Bubbles bad. Very large. Peculiar odor. Tougher

(B)

Lacs containing more or less rosin bubble very badly + bind at 140°

N. C. Condense easily

The shellacs containing rosin fluid at 125-30 but bubble petty badly.

R+P Extra 22 #. Very limpid at 125°. Of heated to 135° and allowed to cool to 125° + foamed. Usual bubbles, Difference #1 21 #. Same as (C).

(C)

(D)

Double A G 21 #. Same as (C)

(E)

R+P in a heart: 17½ No difference
(F)

Diamond R+P: 18½ No difference
(G)

T.N. Pure 15½ ¢: No difference
(A)

Circle R+P 19 ¢: No difference
(J)

R+P Superior: 19½ No Difference
(K)

R+P Special: 21¾: No difference
(L) If anything slightly more bubble

D.C. 22½ ¢: No Difference
(M)

V.S.O. 22 ¢: No Difference
(N)

#1 Button Lac Pure: 19 ¢ Bubbles like
(O) Garnet lac, quite large. Has to be kept at 135° at least to keep fluid. Very tough

#1 Tongue Tongue Button: 18½ ¢
(P) This showed up much better than the Garnets or Other Button. Heat to 140-145 + Cool to 130 while stirring and very few bubbles comparatively are formed.

#1 Button Lac: 18½ ¢ By far the
(S) best as to bubble when heated to 150° + allowed to cool to 130° and pured. Very limpid. This lac was kept at 135-140° C for 1 hr in perfectly limpid condition with hardly a bubble, at the end of which time it showed no sign of condensation. This was true both with and without zinc stearate. At first a little water comes off but soon it fuses quietly without any apparent decomposition.
On keeping at 150° C (300° F) the regular working temp. many more bubbles are formed and the mass condenses in ½ hr.

Made following modifications with #1 Button Lac.

I

9 gms Lac — Toughest and
4 gms tetra — Best
0.5 gm Stearate

II

9 gms lac
5 gms tetra
0.5 gms stearate

III

9 gms lac
6 gms tetra
0.5 gms stearate

IV

9 gms lac
4 gms tetra
1 gm stearate

V

9 gms lac
4 gms tetra
1.5 gms stearate (Excess)

Stearate in excess causes a great many bubbles.
On remelting and keeping below 140° absolutely no bubble appeared in I, II, III. Few in IV and many in V.

Following 6 samples of lacs just arr. May 5. Put thro same test as previous ones.

New York Shellac Co.

Button: Very fair. Not so many bubbles after heating few minutes. 135-140°

No. 1: Just about same as regular. Possibly a shade less bubbling.

No. 2: Same as regular. } Possibly not quite as much decomposition
No. 3: Same as regular. }

V.S.O: Same as regular.

Pure T.N: Same as regular.

Washed # 1 Button lac (R. P. & Co) 1 day in boiling water. Filtrate on evaporation & purification gave a light yellow resin sol. in hot water. This resin decomposed immediately on steam plate, forming a crumbling mass.

On heating over flame it carbonized with an odor like burning rubber.

The lac resulting from the washing process melts without a single bubble, after the water has been driven off. Should be kept at 135° C. (275° F.) for best results.

Determination of Wax in #1 Button Lac (R+P)

wt tube + lac	15.3689
" " − lac	13.4638
" lac	1.9051
Wt. Beaker + wax	45.1652
" "	45.0765
" Wax	00.0887

% Wax 4.65

The lac was saponified with C.P. Borax filtered cold + residue washed with cold water and cold alcohol. The wax was then dissolved from the filter with boiling benzol into a tared beaker, evaporated, dried and weighed.

The purified residue from the aqueous extract of #1 Button Lac was soluble as follows:

1. Completely in boiling water, causing cloudiness on cooling when conc.
2. Partly in NaOH sol., white residue.
3. Almost insol. in alc. hot & cold.
4. Partly in benzol (Hot).

The aqueous sol. is ppted by acids as is also the NaOH sol, making it probable that the substance is of a resinous nature.

Made up a large batch (1-2 lbs) of washed #1 Button Lac. Boiled 1, 2 hrs with three changes of water, put thro the rolls, powdered, and dried.

Quant. detern. H_2O soluble matter in #1 Button lac.

Boiled 12 hrs under reflux with three changes of water.

wt tube +	37.2300
" " —	28.2700
" lac used	8.9600

On evap. the washings to dryness a resinous substance looking like lac remained but having an odor suggesting vanillin. On boiling dry with water, part went into solution & part remained as a dark resin with the mechanical impurities.

A. Water soluble

wt beaker + residue	71.7550
" "	71.4218
" residue	00.3332

B. Water insol.

wt beaker + residue	44.2990
" "	44.1295
" residue	00.1695

Since both A & B were sol. & passed thro the filter after the second extraction, the sum of the two residues A + B = the total matter originally extracted from the lac by water =

.3332
.1695
.5027

% H_2O soluble = 5.61

This represents the net loss in wt. due to extraction with H_2O.

Made up a large batch of tetra-lac

3 lbs washed Button
1 lb tetra
1.6 oz stearate
100 mgms Victoria Blue.

The V. Blue made the resultant mass a bright green so added 100 mgms Basic Violet which changed it to the required blue. As this mix seemed too soft, separated out 2 parts of 2 lbs each and to 1 part added 8 ozs more lac, making it 4 to 1. This blackened the color considerably

the other two lbs left as they were (3-1) for trial. It was more elastic than soft as it was difficult to scratch with the nail but bent like celluloid. Cut like horn. The 4-1 was just as fluid and considerably harder.

Working with Pettit.

Made 10 records from 3-1 and 10 from 4-1. Very hot day (92° F) and both these proportions proved to be too soft. They would not contract from the matrix enough with ordinary cooling. They cemented to the matrix so tightly that when pressure was applied they enclosed air. Tried oiling matrix with amber oil and neats foot oil. N.F. best but process impractical as at causes lint to stick to matrix.

Best results obtained by shortening the blank & making it quite plastic. This allowed air to squeeze out before the wax sealed the outlet.

D-51

Made up 10 records from Button Lac using 5-1

345 gms lac
69 gms tetra
2 gms stearate
Basic Violet

This was very hard. It shrank out of the matrix without difficulty using fan for cooling. Formed 1 blister on the ten records.

S-31

Made up ten records using Rogers Pyatt T N Pure 5½ washed & dried

450 gm lac
150 gm tetra
13½ gm stearate
1½ gm Violett

High temp necessary. Drags badly on coating machine. On pressing, these records

should work better than B 5-1. Then reproducing surface was also better

SA 3-1

Treated TN with alc in cold, filtered, poured into an excess of acidulated water and boiled till ball coag. Decanted and boiled again. Repeated 3 times, boiling 12 hrs in all. Very hard lac having no odor of alcohol resulted. Mixed by a 3-1 regular batch and made into records. This dope crystallized almost instantly, it dragged badly on coating and appeared to contain small particles of condensed dope or dirt. The crystallization an advantage as it became extremely hard.

S 2.5-1

Made a batch with washed TN
2½ parts lac
1 part tetra
as follows

A (Best)

22 g lac
8 g 22 gms tetra
20 gms stearate
½ gm Violet (should have been about 1½ gms as color was too light)
Made into records. Fine
85% good.

Made up a large batch with washed TN
5 lbs lac
2 lbs tetra
10 gms stearate
6 gms violet

B

Burn. The shellac was probably partly condensed by the washing in the wash boiler as it stuck to the sides badly.
Could not filter as it was too thick. Broke two screens on big pot. Condensed solid on reheating. Repeated same formula with small pot. One half could be sucked thru the screen. On reheating the other half it condensed. Finally

Obtained a batch by careful heating + grinding, filtering but it condensed in the coating machine after 12 records were made.

Will heat using I N washed as follows in enamelled pots.

Boil gently with asbestos mat under pot for 6 hrs. Changing the water every hour And stirring frequently. Ground in mill and let dry in the open air

Made up a batch C.

2x { 220 gm lac; 88 gm 22 gm cryst tetra.; 20 gm stearate; 2 gm violet }

Very difficult to filter as the shellac contains so much dirt that it clogs the gauze after about ½ has gone thro. This necessitates reheating the other half and using another filter. Boil this batch towards was much more fluid and did not show any signs of condensation.

Made a large batch of crystallized tetra from benzol. Centrifuged the crystals + dried in the air. The mother liquor contained a lot of oily matter, probably mono-chlor naphthalene. In practice this could be returned to the chlorinating still.

Pettit has evolved a new scheme which obviates the melting of the shellac entirely in the coating machine.

The dope is reduced to 80° mesh and the matrix made very hot. The powd. dope is then introduced into the matrix and rolled till there is a uniform layer on the inside. Hot matrix is then spun and while spinning the hot wax blank is poured in. The trouble with regular dope

was that the tetra separated on the surface of the record forming pits.

I made a dope

D

6 lac
½ tetra crypt
½ stearate.

which gave very good results, no tetra separating.

The batch # marked C was made up with records of which 80 were made from the whole batch. ~~Same~~ It seems that in larger quantities the dope turns out a shade soft, so made up a batch

E

2 x { 22 oz lac T.N
8 oz 22 gm crypt Tetra
5 gm stearate
2½ gm violet

Same trouble in filtering. Batch C made a fine smooth set of blanks and from the appearance of the dope at end of run, could have proceeded indefinitely to coat blanks.

Pettit made the very important discovery that blisters were due to particles of condensed dope embedded in the surface of the wax blank. We cut often a great number of blistered records and in each case found a rubbery brown mass under the blister. This, being elastic tended to force a small area of the shellac dope outward when the pressure was removed. Mr. Edison was fully convinced that this was the cause of the blisters and immediately ordered new blanks made from new material.

On pressing the blanks from Dope E we obtained no blisters with the exception of 2 very peculiar air bubbles which formed over night under the coatings of

two records. They formed blisters each about ⅛ inch in diameter and of a nature never observed before. Possibly due to greasy finger marks on the core. We also discovered that in the coating, the blank must not be burnished against the gate lip so it forms a gathering of latex which deposits on the coat on a sponge that cannot be pressed out. If however, the blank be swung out immediately on closing the gate, no such trouble occurs and from experience we find that a rough coated record gives just as good a reproducing surface as a burnished blank.

Had a small vacuum melting pot made with 3 screens 50, 100, 150 mesh. These screens for removing the muck more effectively without clogging, as shellac used is very dirty. Worked well, the different sizes of dirt particles being plainly visible on the different screens. A large quantity of large particles were deposited on the 50 mesh.

Will have regular pot made this way.

Tried filtering by melting up the ingredients in a double steam heated pot, the bottom of the inner pot being 100 mesh screen. When fluid the inner pot was slowly drawn up and after about 15 min most of the dope had passed through. The scheme fails because it was necessary to heat the dope about ¾ of an hour to get it liquid enough for filtration and as it takes about 15 min for the dope to pass the screen, the dope that has already gone through becomes over heated before it can be poured.

Made the experiment of coating a polished montan blank with a very heavy alc. solution of shellac, drying carefully, baking 12 hrs. and dressing. Good results may be obtained! Push this.

Made a batch of dope with washed TN

22 oz lac
10 oz cryst Tetra
5 gm stearate
3 gm violet

F.

Strained thru 3 screens. Worked fine.

This dope very liquid and works well. We tried a new method of coating with view to getting rid of blisters formed by bridging over. We first dressed the blanks with montan and then gave them a very thin coat of dope. This coat was then flamed, thus breaking open all the air bubbles formed by the air occluded in the surface of blank. We then gave them the regular thickness of dope, flamed, baked and pressed. Not a blister in the lot (Hawk). Only trouble appeared to be particles of montan mechanically picked up by blank before putting in matrix, and smear due to too long burnishing. Possibly this smear also due to insufficient stirring. (Pettit gave orders for a triple stirrer).

We also obtained numerous blind spots caused by the expander corking the ends of blank too soon and thus excluding air. (Can fix)

Aside from this records were alright. The color was a little too light. Will remedy by adding a little nigrosine to next batch.

Wy-Angle

Efperiments to get Rid of Smear:

#1.

6 oz lac (Washed + dried TN)
2½ oz tetra (cryst.)
2 gms stearate
2 gms diferry Canine (+ violet)
No difference. Smear appears on burnishing

#2

6 oz lac
2 oz stearate
½ oz tetra
+ Violet
Absolutely no smear on burnishing
molds well. Too soft and crumbly
under finger nail. Seems to cement
steel to blank better than anything
yet found. No bridging over, very
liquid. No pinholes or flaming.

#3

6 oz lac
1½ oz stearate
1 oz tetra
+ violet
Not enough difference in hardness
to make practical.

#4

8 oz lac
1 oz stearate
2½ oz tetra
+ violet

#5

8 oz lac
½ oz stearate
2½ oz tetra
+ violet

#6

Used 8 oz F dope in pot to which added
⅜ oz stearate. On long burnishing
before the gate, the blank came
out perfect, the only smear being
on the surface which may be
easily wiped off.

Made batch as follows:

2 oz lac
10 g tetra
½ oz stearate
3 gm violet
2 gm Spirit black } good color.

G.

Coated best yet.
Very fine working qualities
Smear still persists - but can be wiped off easily.
Little too soft.

Made batch as follows

2 oz lac
10 g tetra
10 gm stearate
5 gm violet

H

Best blanks yet. Seems to get rid of smear in such large amounts as before
Pressed out beautifully (12 records)
Coated 1 blank leaving it matt surface. Pressed out just as good as the rest which were lustre finished.

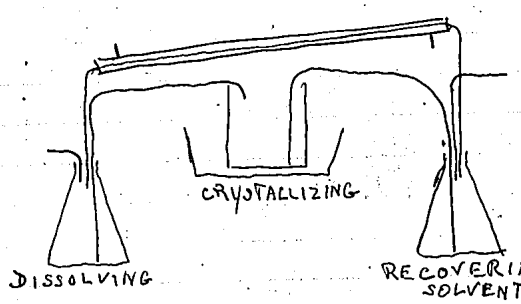
"Mr Edisons suggestion: "melt the dope in the pot, put in the screens and the other pot, invert and keep warm to see if dope wont go through screens by itself. Tried this and dope all went through but it had to be kept so hot for such a length of time that the filtered material was condensed. Tried covering the pots with asbestos to keep in heat + so obviate heating while filtering but it would not go through.

Received instructions from Mr E. to layout a plant for making 1200 lbs dope a day and a plant for crystallizing tetra.

Made a model plant for crystallizing tetra from C Clay

(next page)

#



Found that 50 cc CCl_4 dissolve 300 gms crude tetra, increasing the volume from 50 cc to 260 cc = 42% by volume.

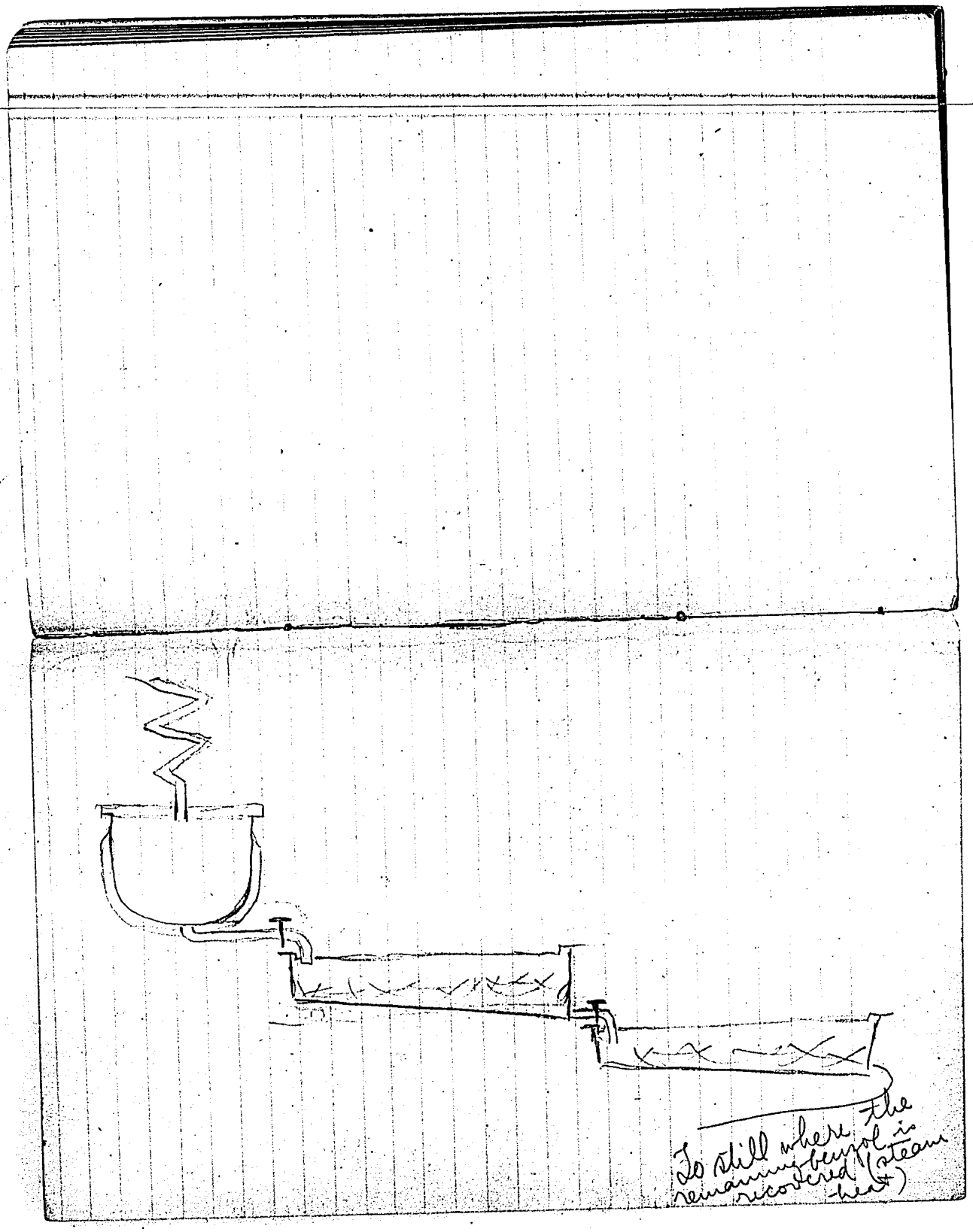
Had a hell of a time with it dope. Couldn't make more than 25 records without it condensing. Only explanation is that the boiling in water partly condenses the shellac. Consequently made following

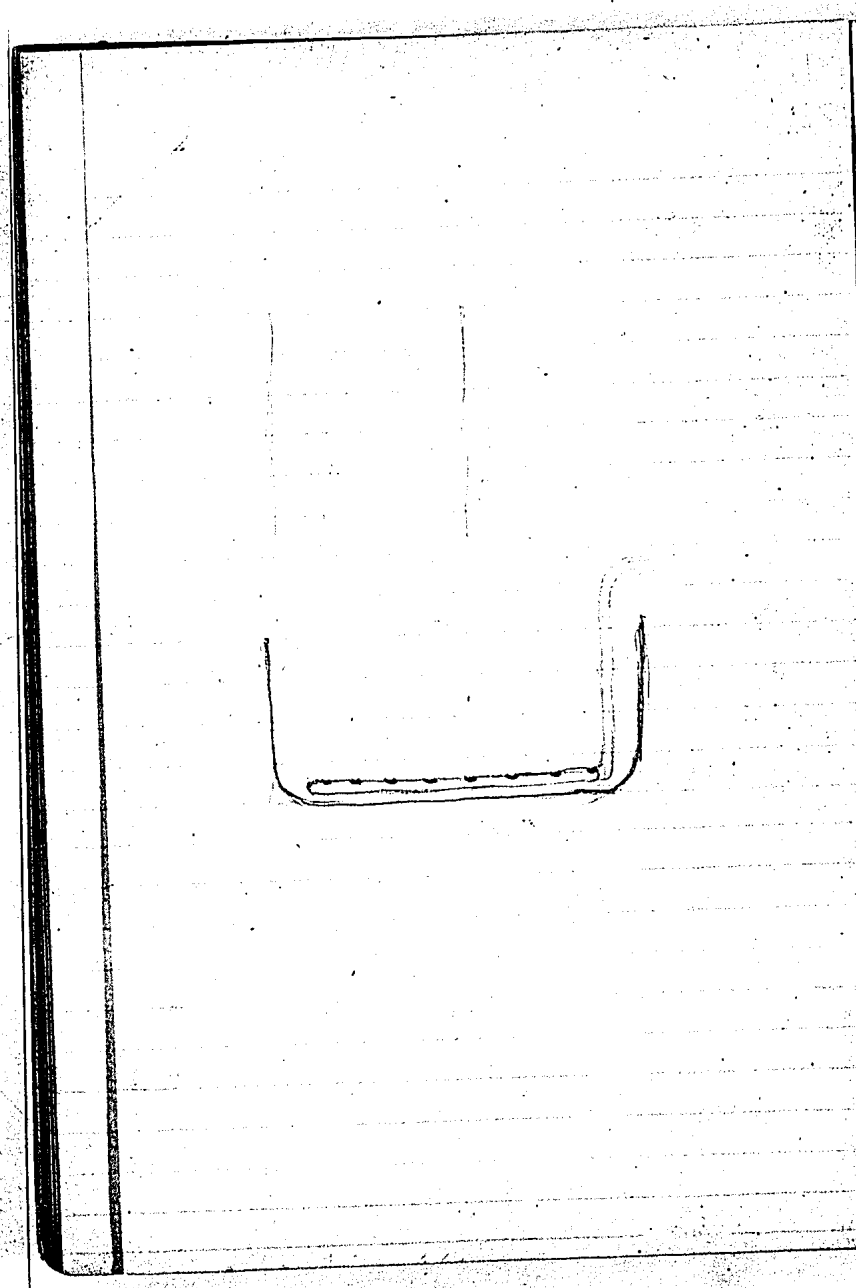
#1.
22 oz unboiled TN
10 oz cryst tetra
10 gm stearate
5 gm violet
5 gm α-naphthylamine

#2.
22 oz TN
10 oz cryst tetra
10 gm stearate
5 gm violet

#3.
22 oz TN
10 oz regular tetra
10 gm stearate
5 gm violet

Works fine at 270° F. Coated 34 blanks and last looks good as first. Must be careful to keep temp between 265-275.


To still where the remaining vapor is recovered (steam heat)



W. water 200 F
Began to come over 300 F

3

3 / 48 / 5 / 240

64 656

4 / 6 / 24

4 165 gr / 16

1 / 16) 1000 / 96 / 40 (6.2

.10 / 6 / 20 / 6 / 1.2

20 grm
1.2 grm lac
2.4

1st crop
Distill off half benzol from filtrate
2nd crop
Distill off half benzol
3rd crop
Distill off all benzol & return residue to
Chrome stills

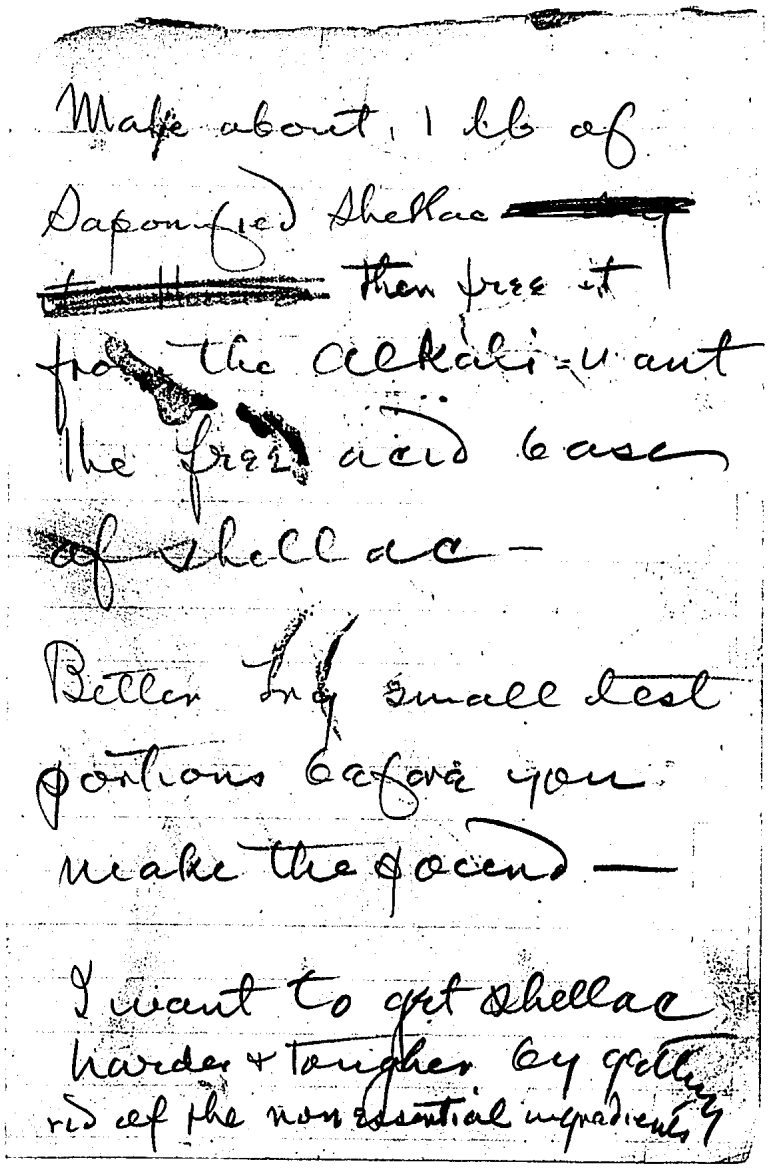
Shellac Wax
50% ceryl + myricyl alc.
50% stearic, palmitic & oleic esters of same
About 20% of the raw shellac

The resin acid exists as a monobasic acid. On boiling with NaOH for 2 hrs it is converted into a tribasic acid. (Benedict + Ulzer)

1 gm pure shellac acid = .066 gm KOH
1 gm tribasic acid = .204 " "

4 oz shellac
1 y

[ITEM(S) FOUND IN BOOK]



Make about 1 lb of
Saponified Shellac
~~[redacted]~~ then free it
from the alkali = want
the free acid base
of shellac —
Better try smallest
portions before you
make the second —

I want to get shellac
harder + tougher by getting
rid of the non essential ingredients